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PATENT ABSTRACTS OF JAPAN

(11) Publication number : 2001-274016
 (43) Date of publication of application : 05.10.2001

(51) Int.CI. H01F 10/14
 C22C 38/00

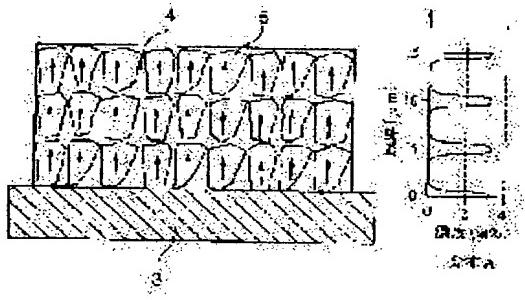
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(54) RARE-EARTH ALLOY FILM MAGNET

(57) Abstract:

PROBLEM TO BE SOLVED: To provide a rare-earth alloy film magnet in which the coarsening of crystal grains is suppressed so as to prevent the disturbance of the c-axes of crystal grains which is caused by the interference between adjacent grains and results in the variation of the direction of magnetization of the magnet when the grains become coarser.

SOLUTION: This rare-earth alloy film magnet has a mixed texture of an Nd₂Fe₁₄B-type crystalline phase in which the c-axes of crystals are oriented in the direction of thickness and an amorphous phase and at least one element selected from among In, Ga, Al, and, Si is added to the film of the magnet in such a way that the concentration of the element is periodically changed in the direction of thickness so that the concentration becomes higher at the portion which is in contact with a substrate. The concentration changing cycle is adjusted to 1–10 µm and the average concentration of the element is adjusted to ≤5 at%. In addition, the amorphous phase is constituted in a ferromagnetic phase.



LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

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CLAIMS

[Claim(s)]

[Claim 1] The rare earth alloy film magnet which adds at least one element in In, Ga, aluminum, and Si in the film, makes this element concentration high in the part which touches a substrate while the c-axis of a crystal has the mixed organization of the Nd₂Fe₁₄B mold crystal phase and amorphous phase which carried out orientation in the direction of thickness, and is characterized by make it change in the direction of thickness periodically.

[Claim 2] The change period of said element concentration is a rare earth alloy film magnet according to claim 1 characterized by being 1-10 micrometers.

[Claim 3] The average concentration of said element is a rare earth alloy film magnet according to claim 1 or 2 characterized by being less than [5at%].

[Claim 4] Said amorphous phase is a rare earth alloy film magnet according to claim 1, 2, or 3 characterized by being ferromagnetism.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the electrical and electric equipment and the rare earth alloy film magnet for electronic equipment, and the rare earth alloy film magnet especially used for a small device or magnetic recording devices, such as a small motor, a microwave oscillator, and a micro machine.

[0002]

[Description of the Prior Art] Although it is indispensable to use a Nd-Fe-B system anisotropic magnet with magnetism powerful as a magnet in order for the need of a minute magnet to

increase with the miniaturization of a device and to obtain high power in recent years, in the process by machining of a sintered magnet, the correspondence to magnetic micrifying is becoming difficult as years pass by, and the limitation of machining is approaching. For this reason, the technique of forming a direct Nd-Fe-B system anisotropic magnet on a substrate by the forming-membranes methods, such as a spatter, and acquiring the last configuration will be examined as a technique in the future.

[0003]

[Problem(s) to be Solved by the Invention] On the occasion of production of the Nd-Fe-B system anisotropic magnet by the forming-membranes method, when thickness is increased, there is a problem to which the mutual intervention of the crystal by coarsening arises and a crystal stacking tendency falls. The turbulence of bearing of a crystallographic axis induces dispersion in the magnetization direction, and a magnetism fall, and by the motor, fluctuation of torque becomes large or it causes the symptom of torque value falling.

[0004] As a solution of the problem about such coarsening, there are some which were indicated by JP,6-151226,A, for example. As this solution is shown in drawing 6, it is the thin film magnet formed in the front face of a substrate 3, and the internal configuration of a thin film magnet has tried to carry out the laminating of the less than 5 micrometers R-Fe-B (rare earth elements in which R contains Y) magnet layer 2, and the metal layer 1 with a thickness of about 10-400A in the direction of thickness by turns, and to prevent coarsening by the metal layer 1. Furthermore, with this solution, the spatter is carried out intermittently, taking the membrane formation quiescent time, by this, the temperature rise by the plasma at the time of membrane formation is eased, and prevention of coarsening is aimed at.

[0005] However, although excelled in the point which can control coarsening comparatively easily, in order that the metal layer 1 which has different-species structure may enter in between, as for the technique of such multilayering in the conventional technique, generating of stress etc. does a bad influence not a little to the magnetic properties of the Nd-Fe-B magnet layer 2. Moreover, by the approach by the above-mentioned intermittent spatter in the conventional technique, since the substrate 3 is heated to hundreds of times, the problem of scaling which arises during a membrane formation pause arises.

[0006] This invention is made paying attention to the trouble which exists in such a Prior art. The place made into the purpose is to offer the rare earth alloy film magnet which controlled big and rough-ization of crystal grain, without causing degradation of magnetic properties.

[0007]

[Means for Solving the Problem] In order to attain the above-mentioned purpose, while the c-axis of a crystal has the mixed organization of the Nd₂Fe₁₄B mold crystal phase and amorphous phase which carried out orientation in the direction of thickness in this invention, at least one element in In, Ga, aluminum, and Si is add in the film, and the element concentration is make high in the part which touches a substrate, and it is characterize by make it change in the direction of thickness periodically.

[0008] Moreover, the change period of said element concentration is set to 1-10 micrometers, make the average concentration below into pentatomic percent (at%), and let an amorphous phase be a ferromagnetic phase further.

[0009] Therefore, in the rare earth alloy film magnet constituted as mentioned above, since an amorphous phase exists between Nd₂Fe₁₄B mold crystal phases, interference of Nd₂Fe₁₄B mold crystal phases was prevented fundamentally, crystal growth to an one direction was made possible, and the high crystal stacking tendency has been realized. Furthermore, since the element of In, Ga, aluminum, and Si which were added in the film promotes amorphous-ization, coarsening is controlled compulsorily. Moreover, if the condition of crystal interference to the first stage like a film formation fault should be formed, the serious bad influence for future crystal stacking tendencies will be done, but by this invention, element concentration added in the part which touches a substrate first is made high, and membranes are formed so that the Nd₂Fe₁₄B mold crystal phase which carried out isolated distribution may be formed into an amorphous host phase. And after that, element concentration is lowered, membranes are formed successingly, growth of a Nd₂Fe₁₄B mold crystal phase is promoted, and the space factor of

this crystal phase is increased. On the other hand, crystal grain originates in the atomic rearrangement to which the growth device mainly happens on a film front face, and the growth rate on the front face of the film becomes the quickest. For this reason, the mutual intervention by the formation of crystal big and rough is first produced at the film topmost part. Since it is such, in this invention, this element concentration is intentionally increased in the phase which reached the thickness which crystal interference produces, the host phase of an amorphous phase is formed again, and isolated distribution of the Nd₂Fe₁₄B mold crystal phase is carried out. This invention presupposes that it is possible to obtain a film magnet with very big thickness by repeating the above procedure, preventing crystal interference.

[0010] If it puts in another way, this invention will have prevented unnecessary coarsening by making high concentration of at least one element in In, Ga, aluminum, and Si in near a substrate front face, and changing it in the direction of thickness periodically. And by sniffing the period of concentration change of this element with 1–10 micrometers, and sniffing the average concentration below with pentatomic percent (at%), the mutual intervention of a crystal was controlled effectively and the high crystal stacking tendency is realized. Furthermore, he is trying to acquire higher magnetism by making an amorphous phase into a ferromagnetic phase.

[0011]

[Embodiment of the Invention] The example of the rare earth metal film magnet concerning the gestalt 1 of the operation to gestalt 1. drawing 1 of operation is shown. In drawing 1, the amorphous phase which a substrate and 4 become from a neodium iron boron nonmagnetic phase in 3, and 5 are Nd₂Fe₁₄B mold crystal phases, like illustration, the interior of a film magnet serves as a mixed organization of the Nd₂Fe₁₄B mold crystal phase 5 and the amorphous phase 4, and each element of In, Ga, aluminum, and Si is added by the distribution of A shown in drawing 1.

[0012] Next, the sample of the gestalt 1 of the operation produced in order to perform a characteristic test is described. First, four kinds per [of In Ga, aluminum, and each Si] element of samples of the gestalt 1 of this operation were produced, and they were prepared so that it might have the element concentration profile of Distribution A which indicated all to drawing 1. When the sample of In addition was produced, specifically, the distribution A of In was given to the interior of a film magnet by performing the coincidence spatter of 2 yuan, having prepared two kinds, Nd–Fe–B and Nd–Fe–B–In, as a target of a spatter, and adjusting the injection power to each target. Ar gas pressure made 4Pa and substrate temperature 560 degrees C. Moreover, the film magnet used the mask together and carried out spatter production by width of face of 1mm, and 100 micrometers of thickness at the lateral surface of an iron cylinder substrate with a diameter of 0.5mm at homogeneity.

[0013] Furthermore, in order to perform the comparison with the conventional thing for the gestalt 1 of operation, the following conventional example 1 and the following conventional example 2 were prepared as a sample of the conventional example. The conventional example 1 carries out spatter membrane formation of the total thickness of 100 micrometers on both sides of about 100A Ti layer for 5 micrometers of every Nd–Fe–B layers, and the conventional example 2 forms Nd–Fe–B, without performing element addition (100 micrometers).

[0014]

[Table 1]

	元素	トルク変動率 (%)	平均トルク (× 10 ⁻⁷ N · m)
実施の形態 1	In	11	1.02
	Ga	10	1.05
	Al	10	1.06
	Si	11	1.04
従来例 1	—	14	0.86
従来例 2	—	25	0.91

[0015] Thus, the sample of the gestalt 1 of this obtained operation and four poles of samples of the conventional example were magnetized, respectively, it included in the motor by having made

this into the magnet rotator, the motor was operated, and torque measurement was performed. Table 1 shows this measurement result. In addition, at the time of measurement, the rate of torque ripple in Table 1 means the rate of the average of this torque to change, and the range of fluctuation of the torque to change, although motor torque is always changed.

[0016] From this measurement result, since torque fluctuation became small from the conventional examples 1 and 2, the sample of the gestalt 1 of the operation which added which element was also understood that control of crystal grain is performed effectively and there is little dispersion in magnetization. Moreover, since the value of average torque became large from the conventional examples 1 and 2, the sample of the gestalt 1 of this operation which added which element was also understood that a crystal stacking tendency is good and magnetism is strong.

[0017] In addition, in the magnet of the gestalt 1 of operation, the Nd₂Fe₁₄B mold crystal phase 5 is good also as a thing containing one or more kinds of rare earth elements other than Nd and transition-metals elements other than Fe other than Nd₂Fe₁₄B. For example, you may be 2 (Nd, Tb) (Fe, Co) 14 B phase, 2 (Nd, Tb) (Fe, nickel) 14 B phase, 2 (Nd, Tb) (Fe, Co, nickel) 14 B phase, 2 (Nd, Tb, Ho) (Fe, Co, nickel) 14 B phase, etc. Moreover, the amorphous phases 4 may be a neodium oxide phase besides a neodium iron boron phase, neodium ****, a neodium boron phase, etc. Moreover, nitrides, such as oxide, such as alloys, such as pure metals, such as others, cobalt and nickel, and titanium, and iron cobalt, iron nickel, quartz glass, and an alumina, and titanium nitride, etc. can be widely used for a substrate 3. [iron]

[0018] The example of the rare earth metal film magnet concerning the gestalt 2 of the operation to gestalt 2. drawing 2 of operation is shown. Although it has a substrate 3, the amorphous phase 4 which consists of a neodium iron boron nonmagnetic phase, and the Nd₂Fe₁₄B mold crystal phase 5 and the interior of a film magnet is the same as that of the case of the gestalt 1 of operation in the point which serves as a mixed organization of the Nd₂Fe₁₄B mold crystal phase 5 and the amorphous phase 4 as this structure is shown in drawing 3. Distribution of at least one element of In, Ga, aluminum, and Si is different in the gestalt 1 of operation at the point added by the distribution of B shown in drawing 2.

[0019] Next, the sample of the gestalt 2 of the operation produced in order to perform a characteristic test is described. Four kinds per [of In, Ga, aluminum, and each Si] alloying element of samples of the gestalt 2 of operation were produced, and they were produced so that it might have the element concentration profile of Distribution B which indicated all to drawing 2. In addition, if this production approach removes the above-mentioned element concentration profile, it is the same as that of the case of the gestalt 1 of operation. Moreover, about the same thing as the conventional examples 1 and 2 indicated at the place of explanation of the sample of the gestalt 2 of the operation obtained by doing in this way, and the gestalt 1 of said operation, like the case of the gestalt 1 of operation, 4 pole magnetization was carried out and it considered as the magnet rotator, and this was included in the motor, it operated, and torque measurement was performed.

[0020]

[Table 2]

	元素	トルク変動率 (%)	平均トルク (× 10 ⁻⁷ N · m)
実施の形態 1	In	10	1.01
	Ga	9	1.04
	Al	10	1.07
	Si	11	1.05
従来例 1	—	14	0.86
従来例 2	—	25	0.91

[0021] Table 2 shows the measurement result. Since torque fluctuation became small from the conventional examples 1 and 2, control of crystal grain is performed effectively and each sample of the gestalt 2 of the operation which added which element was also understood that there is little dispersion in magnetization. Moreover, since the value of average torque became large from

the conventional examples 1 and 2, each sample of the gestalt 2 of the operation which added which element was also understood that a crystal stacking tendency is good and magnetism is strong.

[0022] A substrate is a Ti plate, and the gestalt 3 of gestalt 3. implementation of operation makes concentration distribution of each alloying element of In, Ga, aluminum, and Si half-peak-width [of 0.15 micrometers], and peak concentration 5at% Gaussian distribution, it carries out spatter membrane formation, repeating this a fixed period, and makes it the total thickness of 100 micrometers. In addition, if the gestalt 3 of this operation removes the above-mentioned point, it is the same as the gestalt 1 of operation.

[0023] Next, it attaches and states to the sample produced in order to measure the coercive force per gestalt 3 of this operation. As a sample concerning the gestalt 3 of this operation, eight kinds of things from which a concentration period differs were produced by the plural coincidence spatter about each thing which added each element of In, Ga, aluminum, and Si. In addition, if this production approach removes the point of a substrate ingredient and the element concentration profile to add, it is the same as that of the case of the gestalt 1 of operation.

[0024] Drawing 3 shows the result of having measured coercive force about each [these] sample. As for each sample of the gestalt 3 of operation, this drawing 3 showed that the big coercive force of 400 or more kA/m was acquired for the period of the concentration distribution about an alloying element by 1 micrometers or more. Next, drawing 4 shows the result evaluated about the crystal stacking tendency of each sample. This drawing 4 is the result of calculating psi value (= psi 50) which measures an X diffraction pattern and is set to $I(\psi)/I(0) = 0.5$ from the dependency of the integrated intensity I of the diffraction peak of the acquired field (006), setting the scanning shaft in X-ray diffractometer to theta/2theta, and changing offset angle psi of setting of a sample, and means that a crystal stacking tendency is so high that the value of psi 50 is small. psi 50 is a comparatively low value from this drawing 4 in the range whose period is 1–10 micrometers, and, thereby, any sample concerning the gestalt 3 of operation was understood that a crystal stacking tendency is good.

[0025] A substrate is a Ti plate, the gestalt 4 of gestalt 4. implementation of operation is what carried out spatter membrane formation, having made concentration distribution of each alloying element of In, Ga, aluminum, and Si into having—with half peak width of 1.5 micrometers—fixed peak concentration Gaussian distribution, and repeating this with the period of 5 micrometers, and was made into the total thickness of 100 micrometers, and if it removes these points, the structure of it is the same as the gestalt 1 of operation.

[0026] Next, the sample of six kinds of things from which peak concentration differs was produced by the plural coincidence spatter about the sample which added each element per [In, Ga, aluminum, and Si] gestalt 4 of this operation. In addition, if the production approach of the gestalt 4 operation removes the point of a substrate and an element concentration profile, it is the same as that of the case of the gestalt 1 of operation.

[0027] The result of having measured coercive force about each [these] sample to drawing 5 is shown. Element concentration shows the average concentration in the film among drawing. This drawing showed that average concentration could acquire the good property of 400 or more kA/m of coercive force for any sample concerning the gestalt 4 of operation less than [5at%].

[0028] Let the amorphous phase 4 be a neodium iron boron ferromagnetism phase by adjusting the presentation of a Nd-Fe-B target in the internal configuration of the film magnet concerning the gestalt 1 of gestalt 5. implementation of operation. Next, the sample concerning the gestalt 5 of the operation produced in order to measure the torque about the motor using the rotator produced by the gestalt 5 of this operation is described. The sample concerning the gestalt 5 of the produced operation is what set the alloying element to Si, and made concentration distribution of this Si the distribution curve of A indicated by drawing 1. Moreover, if the production approach of this sample removes the point that an element concentration profile and the amorphous phase 4 are neodium iron boron ferromagnetism phases, it is the same as that of the case of the gestalt 1 of operation.

[0029]

[Table 3]

	トルク変動率 (%)	平均トルク ($\times 10^{-7}$ N · m)
実施の形態5	10	1.35
参考 (実施の形態1)	9	1.04

[0030] Thus, four poles of each sample of the gestalt 5 of the obtained operation were magnetized, and it considered as the magnet rotator, and included in the motor, this motor was operated, and torque measurement was performed. Table 3 shows this result. In addition, the result about the thing of the alloying element Si in the gestalt 1 of operation is also doubled and shown in this table for the comparison. Since this operation gestalt 5 made the amorphous phase 4 the ferromagnetic phase, this table 3 showed it, although it could acquire bigger average torque than the gestalt 1 of operation.

[0031]

[Effect of the Invention] Since this invention is constituted as mentioned above, it does the following effectiveness so. While the c-axis of a crystal has the mixed organization of the Nd₂Fe₁₄B mold crystal phase and amorphous phase which carried out orientation in the direction of thickness according to invention according to claim 1 to 4 Since at least one element in In, Ga, aluminum, and Si was added in the film, and this element concentration was made high in the part which touches a substrate and it was made to change in the direction of thickness periodically, big and rough-ization of crystal grain is prevented, and a property with little [a crystal stacking tendency is high and] dispersion in magnetization can be realized. Therefore, if the rare earth alloy film magnet of this invention is used for a motor, torque fluctuation of a motor can be made small.

[0032] Moreover, according to invention according to claim 2, since the change period of said element concentration is 1–10 micrometers, in addition to said effectiveness, the mutual intervention of a crystal can be controlled effectively and a high crystal stacking tendency can be realized.

[0033] Moreover, according to invention according to claim 3, since the average concentration of said element is less than [5at%], in addition to said effectiveness, the mutual intervention of a crystal can be controlled effectively and a high crystal stacking tendency can be realized.

[0034] Moreover, according to invention according to claim 4, since an amorphous phase is ferromagnetism, in addition to said effectiveness, higher magnetism can be acquired.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] It is a sectional view for explaining the film magnet concerning the gestalt 1 of operation of this invention.

[Drawing 2] It is a sectional view for explaining the film magnet concerning the gestalt 2 of operation of this invention.

[Drawing 3] It is the property Fig. of the coercive force about the film magnet concerning the gestalt 3 of operation of this invention.

[Drawing 4] It is the property Fig. of the crystal stacking tendency about the film magnet concerning the gestalt 3 of operation of this invention.

[Drawing 5] It is the property Fig. of the coercive force about the film magnet concerning the gestalt 4 of operation of this invention.

[Drawing 6] It is a sectional view for explaining the conventional film magnet.

[Description of Notations]

1 A metal layer, 2 A R-Fe-B magnet layer, 3 A substrate, 4 An amorphous phase, 5 Nd-Fe-B mold crystal phase.

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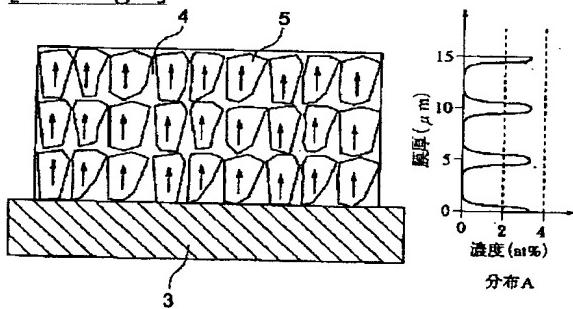
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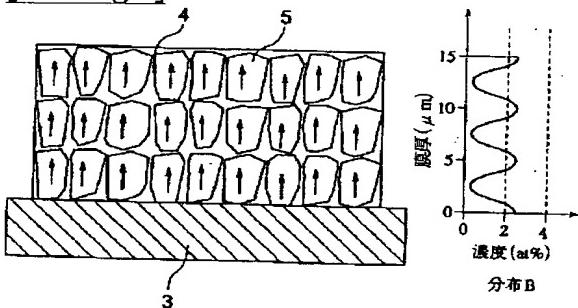
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DRAWINGS

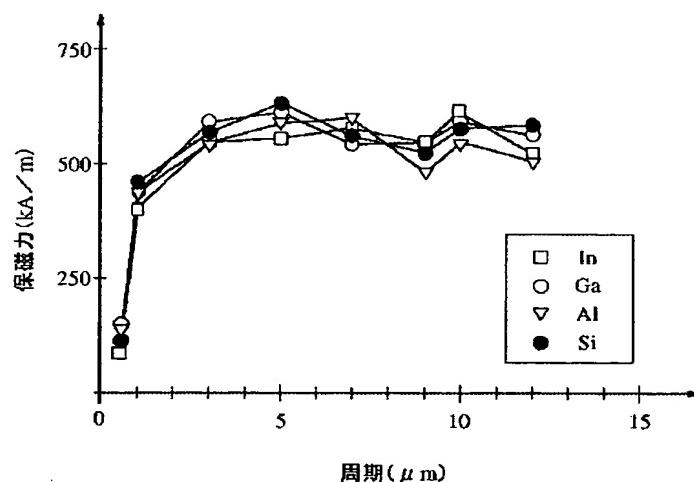
[Drawing 1]



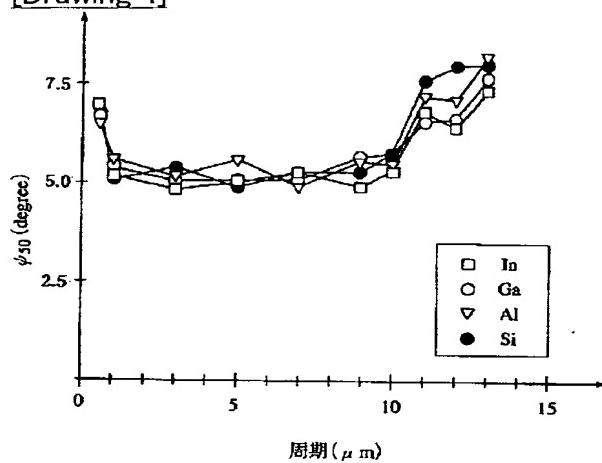
[Drawing 2]



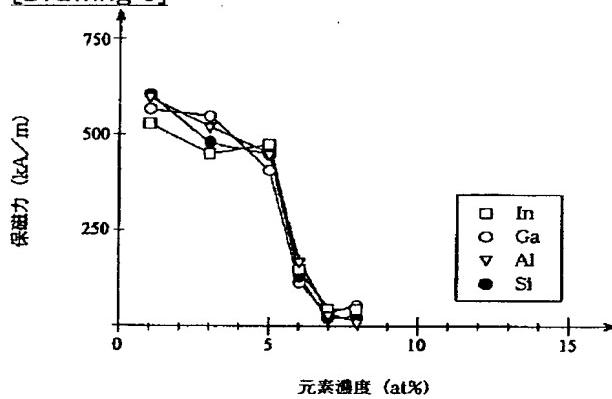
[Drawing 3]



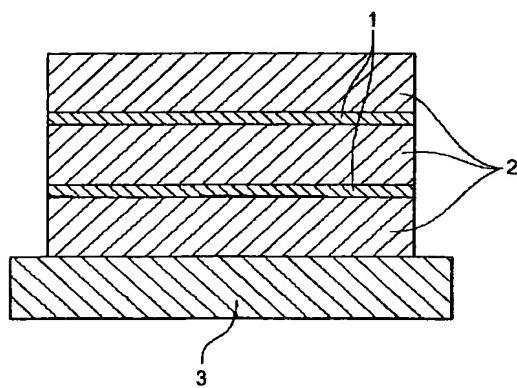
[Drawing 4]



[Drawing 5]



[Drawing 6]



[Translation done.]

(19) 日本国特許庁 (JP)

(12) 公開特許公報 (A)

(11) 特許出願公開番号

特開2001-274016

(P2001-274016A)

(43) 公開日 平成13年10月5日 (2001.10.5)

(51) Int.Cl.⁷
 H 01 F 10/14
 C 22 C 38/00

識別記号
 3 0 3

F I
 H 01 F 10/14
 C 22 C 38/00

テマコード(参考)
 5 E 04 9
 3 0 3 D

審査請求 未請求 請求項の数4 O.L (全6頁)

(21) 出願番号 特願2000-84215(P2000-84215)

(22) 出願日 平成12年3月24日 (2000.3.24)

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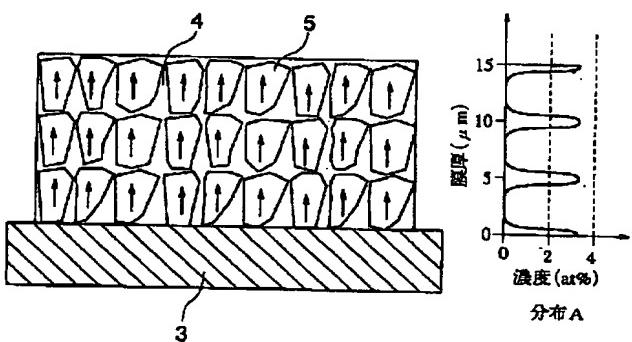
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(54) 【発明の名称】 希土類合金膜磁石

(57) 【要約】

【課題】 希土類系膜磁石の厚膜化に伴う結晶粒粗大化は、隣接する粒子との干渉を引き起こし、その結果、結晶のc軸が乱れて磁化方向にばらつきが生じてしまう。これを改善するため、結晶粒の粗大化を抑制した希土類合金膜磁石を提供する。

【解決手段】 膜厚方向に結晶のc軸が配向したNd₂Fe₁₄B型結晶相と非結晶相の混合組織を有し、膜中にIn、Ga、Al、Siのうちの少なくとも1つの元素を添加し、この元素濃度を基板に接する部分で高くし、かつ、膜厚方向に周期的に変化させる。この元素濃度の周期は1~10μmとし、元素の平均濃度は5at%以下とする。また、非結晶相を強磁性相とする。



【特許請求の範囲】

【請求項1】 膜厚方向に結晶のc軸が配向したNd₂Fe₁₄B型結晶相と非結晶相との混合組織を有するとともに、膜中にIn、Ga、Al、Siのうちの少なくとも1つの元素を添加し、この元素濃度を、基板に接する部分で高くし、かつ、膜厚方向に周期的に変化させたことを特徴とする希土類合金膜磁石。

【請求項2】 前記元素濃度の変化周期は1～10μmであることを特徴とする請求項1記載の希土類合金膜磁石。

【請求項3】 前記元素の平均濃度は5at%以下であることを特徴とする請求項1または2記載の希土類合金膜磁石。

【請求項4】 前記非結晶相は強磁性であることを特徴とする請求項1、2または3記載の希土類合金膜磁石。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】 この発明は電気および電子機器用の希土類合金膜磁石、特に、小型モータ、マイクロ波発振器、マイクロマシン等の小型デバイスあるいは磁気記録デバイスに用いる希土類合金膜磁石に関するものである。

【0002】

【従来の技術】 近年、デバイスの小型化に伴い微小磁石の需要が増しており、高出力を得るために、磁石としては磁力の強いNd-Fe-B系異方性磁石を用いることが不可欠となっているが、焼結磁石の機械加工による製法では、年を追うごとに磁石の微小化への対応が難しくなってきており、機械加工の限界が近づいている。このため、将来技術として、スパッタなどの成膜法により基板上に直接Nd-Fe-B系異方性磁石を形成して最終形状を得る手法が検討されている。

【0003】

【発明が解決しようとする課題】 成膜法によるNd-Fe-B系異方性磁石の作製に際し、膜厚を増大させると結晶粒粗大化による結晶の相互干渉が生じて結晶配向性が低下する問題がある。結晶軸の方位の乱れは磁化方向のばらつきや磁力低下を生み、モータではトルクの変動が大きくなったり、トルク値が低下する等の症状を引き起こす。

【0004】 このような結晶粒粗大化に関する問題の解決策としては、たとえば、特開平6-151226号公報に記載されたものがある。この解決策は、図6に示すように、基板3の表面に形成された薄膜磁石であって、薄膜磁石の内部構成は、膜厚方向に5μm未満のR-F_e-B(RはYを含む希土類元素)磁石層2と厚さ約10～400オングストロームの金属層1とが交互に積層されたものであって、金属層1により結晶粒粗大化を防止することを試みている。さらに、この解決策では、成膜休止時間をとりながら間欠的にスパッタしており、こ

れによって成膜時のプラズマによる温度上昇を緩和し、結晶粒粗大化の防止を図っている。

【0005】 しかし、従来技術におけるこのような多層化の手法は、結晶粒粗大化を比較的簡単に抑制することができる点では優れているが、異種構造を有する金属層1が間にいるため、応力の発生等、Nd-Fe-B磁石層2の磁気特性に対して少なからず悪影響を及ぼす。また、従来技術における上記間欠スパッタによる方法では、基板3が数百度まで加熱されているため、成膜休止中に起こる表面酸化の問題が生じる。

【0006】 本発明は、このような従来の技術に存在する問題点に着目してなされたものである。その目的とするところは、磁気特性の劣化を引き起こすことなく結晶粒の粗大化を抑制した希土類合金膜磁石を提供することにある。

【0007】

【課題を解決するための手段】 上記目的を達成するために、本発明では、膜厚方向に結晶のc軸が配向したNd₂Fe₁₄B型結晶相と非結晶相との混合組織を有するとともに、膜中にIn、Ga、Al、Siのうちの少なくとも1つの元素を添加し、その元素濃度を基板に接する部分で高くし、かつ、膜厚方向に周期的に変化させたことを特徴とする。

【0008】 また、前記元素濃度の変化周期を1～10μmとし、その平均濃度を5原子パーセント(at%)以下とし、さらに、非結晶相を強磁性相とする。

【0009】 したがって、上記のように構成された希土類合金膜磁石においては、Nd₂Fe₁₄B型結晶相間に非結晶相が存在するので、基本的にNd₂Fe₁₄B型結晶相同士の干渉を防いで、一方向への結晶成長を可能にし、高い結晶配向性を実現している。さらに、膜中に添加されたIn、Ga、Al、Siの元素が非結晶化を促進するので、結晶粒粗大化が強制的に抑制される。また、万一膜形成過程の初期に結晶干渉の状態が形成されてしまうと、以後の結晶配向性に重大な悪影響を及ぼすが、本発明では、まず基板に接する部分において添加した元素濃度を高くし、非結晶母相中に孤立分散したNd₂Fe₁₄B型結晶相を形成するよう成膜している。そして、その後、元素濃度を下げて引き続き成膜して、Nd₂Fe₁₄B型結晶相の成長を促進し、この結晶相の占積率を増加している。一方、結晶粒は、その成長機構が主に膜表面で起る原子再配列に起因しており、膜表面での成長速度が最も速くなる。このため、結晶粗大化による相互干渉は最初に膜最上部において生じる。このようしたことから、本発明では、結晶干渉が生じる膜厚に達した段階で該元素濃度を意図的に増加し、再び非結晶相の母相を形成してNd₂Fe₁₄B型結晶相を孤立分散させていく。本発明は、以上の手順を繰り返すことにより、結晶干渉を防止しつつ非常に大きな膜厚を持つ膜磁石を得ることが可能とするものである。

【0010】換言すれば、本発明は、In、Ga、Al、Siのうちの少なくとも1つの元素の濃度を、基板表面付近において高くし、膜厚方向に周期的に変化させることにより、不要な結晶粒粗大化を防止している。そして、該元素の濃度変化の周期を1~10μm、その平均濃度を5原子パーセント(at%)以下とすることにより、効果的に結晶の相互干渉を抑制し、高い結晶配向性を実現している。さらに、非結晶相を強磁性相とすることにより、より高い磁力を得るようにしている。

【0011】

【発明の実施の形態】実施の形態1. 図1に実施の形態1に係る希土類金属膜磁石の例を示す。図1において、3は基板、4はネオジウム鉄ボロン非磁性相からなる非結晶相、5はNd₂Fe₁₄B型結晶相であり、図示のごとく膜磁石の内部はNd₂Fe₁₄B型結晶相5と非結晶相4との混合組織となっており、In、Ga、Al、Siの各元素が図1中に示されたAの分布で添加されているものである。

【0012】次に、特性試験を行うために作製した実施の形態1のサンプルについて述べる。まず、本実施の形態1のサンプルは、In、Ga、Al、Siそれぞれの*

	元素	トルク変動率(%)	平均トルク($\times 10^{-7}$ N·m)
実施の形態1	In	1.1	1.02
	Ga	1.0	1.05
	Al	1.0	1.06
	Si	1.1	1.04
従来例1	-	1.4	0.86
従来例2	-	2.5	0.91

【0015】このようにして得られた本実施の形態1のサンプルおよび従来例のサンプルをそれぞれ4極着磁し、これを磁石回転子としてモータに組み込み、モータを運転してトルク測定を行った。表1はこの測定結果を示す。なお、表1におけるトルク変動率とは、測定時モータトルクは常に変動しているが、この変動するトルクの平均値と変動するトルクの変動幅との割合を意味する。

【0016】この測定結果より、いずれの元素を添加した実施の形態1のサンプルも、従来例1、2よりトルク変動が小さくなることから、結晶粒の抑制が効果的に行われており、磁化のばらつきの少ないことがわかった。また、いずれの元素を添加した本実施の形態1のサンプルも、従来例1、2より平均トルクの値が大きくなることから、結晶配向性が良好で磁力が強いことがわかった。

【0017】なお、実施の形態1の磁石において、Nd₂Fe₁₄B型結晶相5は、Nd₂Fe₁₄Bのほかにも、Nd以外の希土類元素やFe以外の遷移金属元素を1種類以上含むものとしても良い。たとえば、(Nd、Tb)₂(Fe、Co、Ni)₁₄B相、(Nd、Tb、Ho)₂(Fe、Co、Ni)₁₄B相などであっても良い。また、非結晶相4は、ネオジウム鉄ボロン相の他、ネオジウム酸化物相、ネオジウム鉄相、ネオジウムボロン相などであっても良い。また、基板3は、鉄のほか、コバルトやニッケル、チタンなどの純金属や鉄コバルト、鉄ニッケルなどの合金、石英ガラスやアルミナなどの酸化物、窒化チタンなどの窒化物などを広く用いることができる。

【0018】実施の形態2. 図2に実施の形態2に係る希土類金属膜磁石の例を示す。この構造は、図3に示されるように、基板3、ネオジウム鉄ボロン非磁性相からなる非結晶相4、Nd₂Fe₁₄B型結晶相5を有し、膜磁石の内部はNd₂Fe₁₄B型結晶相5と非結晶相4との混合組織となっている点において実施の形態1の場合と同様であるが、In、Ga、Al、Siの少なくとも1つの元素の分布が、図2中に示されているBの分布で添加されている点で実施の形態1とは相違する。

【0019】次に、特性試験を行うために作製した実施の形態2のサンプルについて述べる。実施の形態2のサンプルは、In、Ga、Al、Siそれぞれの添加元素

* 元素につき4種類作製し、いずれも図1に記載した分布Aの元素濃度プロファイルを持つように調製した。具体的には、例えば、In添加のサンプルを作製する場合、スパッタのターゲットとしてNd-Fe-BとNd-Fe-B-Inの2種類を用意し、各ターゲットへの投入電力を調節しながら2元同時スパッタを行うことにより、膜磁石の内部にInの分布Aを付与した。Arガス圧は4Pa、基板温度は560°Cとした。また、膜磁石は、マスクを併用して直径0.5mmの鉄製の円柱基板の外側面に幅1mm、膜厚100μmで均一にスパッタ作製した。

【0013】さらに、実施の形態1を従来のものとの比較を行うために、従来例のサンプルとして次の従来例1および従来例2を用意した。従来例1は、Nd-Fe-B層5μmごとに約100ÅのTi層を挟んで総厚100μmをスパッタ成膜したものであり、従来例2は、元素添加を行わずにNd-Fe-Bの成膜(100μm)を行ったものである。

【0014】

【表1】

につき4種類作製し、いずれも図2に記載した分布Bの元素濃度プロファイルを持つように作製した。なお、この作製方法は、上記元素濃度プロファイルを除いては実施の形態1の場合と同様である。また、このようにして得られた実施の形態2のサンプルおよび前記実施の形態1の説明のところに記載した従来例1、2と同一のもの*

	元素	トルク変動率 (%)	平均トルク ($\times 10^{-7}$ N・m)
実施の形態1	In	10	1.01
	Ga	9	1.04
	Al	10	1.07
	Si	11	1.05
従来例1	—	14	0.86
従来例2	—	25	0.91

【0021】表2はその測定結果を示す。いずれの元素を添加した実施の形態2の各サンプルも、従来例1、2よりトルク変動が小さくなることから、結晶粒の抑制が効果的に行われており磁化のばらつきが少ないことがわかった。また、いずれの元素を添加した実施の形態2の各サンプルも、従来例1、2より平均トルクの値が大きくなることから、結晶配向性が良好で磁力が強いことがわかった。

【0022】実施の形態3。実施の形態3は、基板がTi平板であり、In、Ga、Al、Siの各添加元素の濃度分布を半価幅0.15μm、ピーク濃度5at%のガウス分布とし、これを一定の周期で繰り返しながらスペッタ成膜し、総厚100μmとしたものである。なお、この実施の形態3は、上記の点を除いては、実施の形態1と同一である。

【0023】次に、この実施の形態3につき、保磁力を測定するために作製したサンプルについて述べる。この実施の形態3に係るサンプルとしては、In、Ga、Al、Siの各元素を添加したものそれぞれについて、濃度周期の異なるもの8種類を多元同時スペッタ法で作製した。なお、この作製方法は、基板材料と添加する元素濃度プロファイルの点を除いては実施の形態1の場合と同様である。

【0024】図3はこれら各サンプルについて保磁力を測定した結果を示す。この図3から、実施の形態3の各サンプルは、添加元素についての濃度分布の周期が1μm以上で400KA/m以上の大きな保磁力が得られることがわかった。次に、図4は、各サンプルの結晶配向性について評価した結果を示す。この図4は、X線回折装置におけるスキャン軸をθ/2θとし、サンプルのセッティングのオフセット角φを変化させながらX線回折パターンの測定を行って、得られた(006)面の回折ピークの積分強度Iの依存性からI(φ)/I(0)=0.5となるφ値(=φ_{so})を求めた結果であって、φ_{so}の値が小さいほど結晶配向性が高いことを意味する。この図4から、実施の形態3に係るいずれのサンプル

*について、実施の形態1の場合と同様に、4極着磁して磁石回転子とし、これをモータに組み込んで運転してトルク測定を行った。

【0020】

【表2】

も、周期が1~10μmの範囲でφ_{so}が比較的低い値となっており、これにより結晶配向性が良好であることがわかった。

【0025】実施の形態4。実施の形態4は、基板がTi平板であり、In、Ga、Al、Siの各添加元素の濃度分布を半価幅1.5μmで一定のピーク濃度を持つガウス分布とし、これを5μmの周期で繰り返しながらスペッタ成膜し、総厚100μmとしたもので、これらの点を除いては、構造は実施の形態1と同一である。

【0026】次に、この実施の形態4につきIn、Ga、Al、Siの各元素を添加したサンプルについて、ピーク濃度の異なるもの6種類のサンプルを多元同時スペッタ法で作製した。なお、実施の形態4の作製方法は、基板と元素濃度プロファイルの点を除いては実施の形態1の場合と同様である。

【0027】図5にこれら各サンプルについて保磁力を測定した結果を示す。図中、元素濃度は、膜中の平均濃度を示している。この図から、実施の形態4に係るいずれのサンプルも、平均濃度が5at%以下で保磁力400KA/m以上の良好な特性を得られることがわかった。

【0028】実施の形態5。実施の形態1に係る膜磁石の内部構成において、Nd-Fe-Bターゲットの組成を調整することにより、非結晶相4をネオジウム鉄ボロン強磁性相としたものである。次に、この実施の形態5により作製された回転子を用いたモータについてのトルクを測定するために作製した実施の形態5に係るサンプルについて述べる。作製した実施の形態5に係るサンプルは、添加元素をSiとしたもので、このSiの濃度分布を図1に記載されたAの分布曲線とした。また、このサンプルの作製方法は、元素濃度プロファイルおよび非結晶相4がネオジウム鉄ボロン強磁性相である点を除いては実施の形態1の場合と同様である。

【0029】

【表3】

	トルク変動率 (%)	平均トルク ($\times 10^{-7} \text{ N} \cdot \text{m}$)
実施の形態5	10	1.35
参考(実施の形態1)	9	1.04

【0030】このようにして得られた実施の形態5の各サンプルを4極着磁して磁石回転子としてモータに組み込み、このモータを運転してトルク測定を行った。表3はこの結果を示す。なお、同表には比較のため、実施の形態1における添加元素Siのものについての結果をも合わせて示している。この表3から、本実施形態5は、非結晶相4を強磁性相としているため、実施の形態1よりも大きな平均トルクを得ることができるがわかった。

【0031】

【発明の効果】本発明は、以上のように構成されているため、次のような効果を奏する。請求項1～4記載の発明によれば、膜厚方向に結晶のc軸が配向したNd₂Fe₁₄B型結晶相と非結晶相との混合組織を有するとともに、膜中にIn、Ga、Al、Siのうちの少なくとも1つの元素を添加し、この元素濃度を、基板に接する部分で高くし、かつ、膜厚方向に周期的に変化させたので、結晶粒の粗大化が防止され、結晶配向性が高く磁化のばらつきが少ない特性を実現することができる。したがって、本発明の希土類合金膜磁石をモータに用いれば、モータのトルク変動を小さくすることができる。

【0032】また、請求項2記載の発明によれば、前記元素濃度の変化周期は1～10μmであるので、前記効果に加え、効果的に結晶の相互干渉を抑制して高い結晶*

*配向性を実現することができる。

【0033】また、請求項3記載の発明によれば、前記元素の平均濃度は5at%以下であるので、前記効果に加え、効果的に結晶の相互干渉を抑制して高い結晶配向性を実現することができる。

【0034】また、請求項4記載の発明によれば、非結晶相が強磁性であるので、前記効果に加え、より高い磁力を得ることができる。

【図面の簡単な説明】

【図1】本発明の実施の形態1に係る膜磁石を説明するための断面図である。

【図2】本発明の実施の形態2に係る膜磁石を説明するための断面図である。

【図3】本発明の実施の形態3に係る膜磁石についての保磁力の特性図である。

【図4】本発明の実施の形態3に係る膜磁石についての結晶配向性の特性図である。

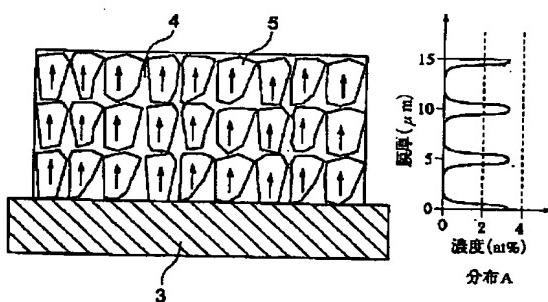
【図5】本発明の実施の形態4に係る膜磁石についての保磁力の特性図である。

【図6】従来の膜磁石を説明するための断面図である。

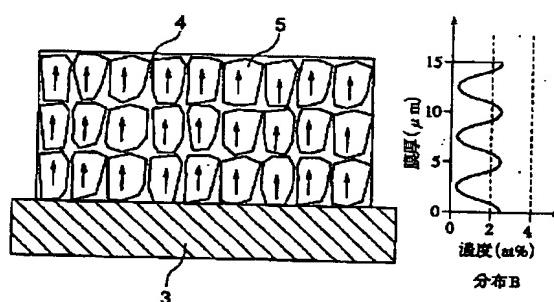
【符号の説明】

1 金属層、2 R-F_e-B磁石層、3 基板、4 非結晶相、5 Nd-F_e-B型結晶相。

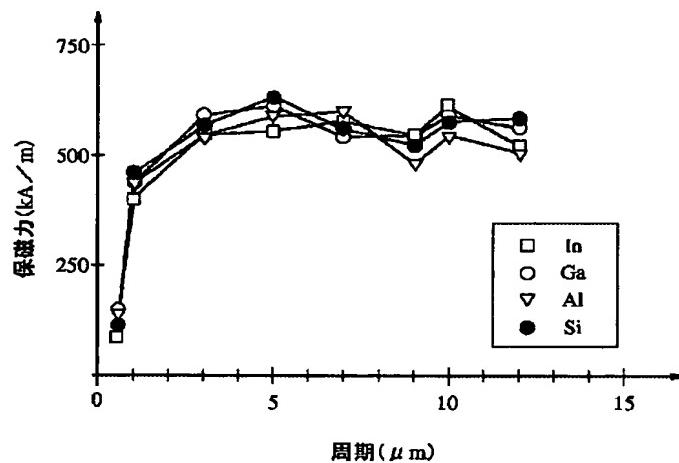
【図1】



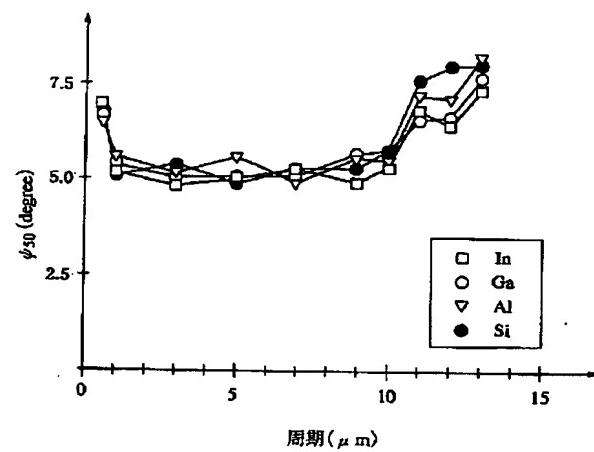
【図2】



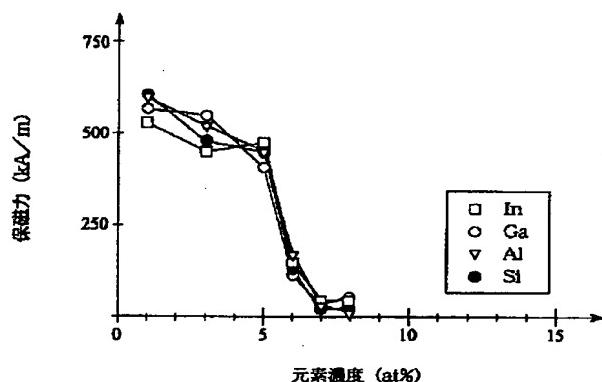
【図3】



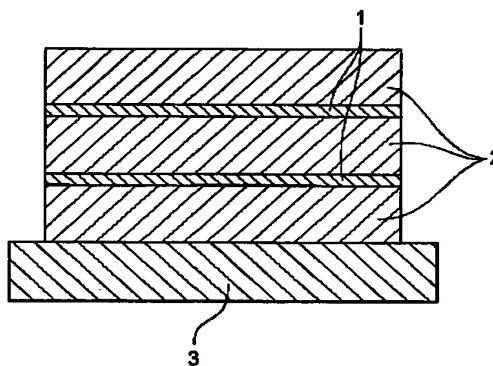
【図4】



【図5】



【図6】



フロントページの続き

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F ターム(参考) 5E049 AA01 AA09 AC01 BA01 BA29
DB02 GC01